

# INFORMATION REPORT

CD NO.

COUNTRY East Germany

DATE DISTR 1 July 1953

SUBJECT Research Projects of the Main Laboratory at Elektrochemisches Kombinat Bitterfeld

NO. OF PAGES 3

PLACE ACQUIRED

NO. OF ENCLS.  
(LISTED BELOW)

DATE OF INFO.

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SUPPLEMENT TO REPORT NO.

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SOURCE

1. The Main Laboratory (Hauptlaboratorium) of Elektrochemisches Kombinat Bitterfeld (EKB) has two departments: the Analytical Department and the Experimental Department (Versuchsabteilung). While the Experimental Department belongs administratively to the Main Laboratory, it receives its research assignments from the Central Research Laboratory (Zentrales Forschungsinstitut) of EKB. The latter office is headed by Dr. Cuenther Mehne. Walter Bunze is head of the Main Laboratory.
2. In 1952 the Experimental Department was engaged in the following research work:
  - a) Working out a new method for the production of oxalic acid. EKB had previously produced oxalic acid from beet sugar and nitric acid. This procedure required the use of large containers of V2A steel, an item which was more and more in short supply. Work on the new method was begun in April 1952 and terminated, on a laboratory scale, in January 1953. Sodium chloride dissolved in water is decomposed in an electrolytic bath containing mercury; the sodium electrolytically released and the mercury amalgamize into sodium-mercury alloy. The alloy, while being continuously stirred, is treated with a stream of carbon dioxide; as a result sodium oxalate is formed. This is dissolved in distilled water and led over wofatit, which acts as ion exchanger. The wofatit is in a pipe. Prior to its use, it is treated with chloric acid. Oxalic acid of a concentration of 99.8 and 99.9 percent is released from the lower end of the pipe, which is in a vertical position. This method not only dispenses with the use of V2A containers but also is considerably cheaper than the old method. Production costs of oxalic acid produced according to the old method amounted to 900 DM/ton; costs of production by the new method have been computed to amount to only about 150 DM/ton. The new method has not yet been applied on a production scale because the 1953 plan makes no funds available for it. It will be applied in 1954.
  - b) During the first part of 1952, the department received a Russian order for the preparation, on a laboratory scale, of highly concentrated selenic acid. Twenty thousand DM were made available for the carrying out of the order. The department had old supplies of selenium metal. Some of it was dissolved in nitric acid of about 65 percent

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concentration. Selenious acid thus formed was oxidized by means of chloric acid; as a result selenic acid was obtained. This was subjected to distillation in a high vacuum container. The result was solid selenic acid of 100 percent concentration. The department produced one kilogram of 100 percent selenic acid. The difficulty of this procedure lay in the oxidation of selenious acid with chloric acid; large amounts of this latter agent were required, and oxidation had to be repeated a great number of times until the desired degree of concentration was reached.

- c) In August 1952, the department received another Russian order for the production of ten kilograms of telluric acid; the order allegedly came from the same source as the order for the preparation of selenic acid. Ten kilograms of tellurium were bought in West Germany for this purpose.<sup>4/</sup> The metal was dissolved in nitric acid to which small quantities of hydrochloric acid had been added. Telluric acid thus formed was concentrated in a vaporizing apparatus where telluric acid of high purity crystallized. Ten kilograms of pure telluric acid and one kilogram of selenic acid were shipped to Russia together.

3. In 1953, the Experimental Department of the Main Laboratory engaged in the following work:

- a) Dechlorination of sodium hydroxide with ammonia under pressure. In December 1952, a Russian order was received from the Lenna works requesting the resumption of research work along this line which had been carried out during 1946 and 1947 on behalf of the Russians.<sup>5/</sup> During that period when the Kombinat belonged to IAG Kaustik, a Russian, Engelin (fnu), supervised the work; when Engelin left, Joffe (fnu), also a Russian, took over. The work was then carried out under the direction of Dr. Udo Ehrhardt. Ehrhardt told his co-workers that the Russians were extremely interested in a well-functioning dechlorination procedure, because chlorine contents in sodium hydroxide seriously hampered the use of this latter agent in the production of artificial silk. The Russians, according to Ehrhardt's story in 1946 and 1947, intended to open the world's largest artificial silk enterprise somewhere in Karelia. They were therefore badly in need of an efficient dechlorination procedure. In 1946 and 1947, Ehrhardt carried out the order first on a laboratory scale and then built a pilot plant which produced small amounts of dechlorinated sodium hydroxide.<sup>7/</sup> The pilot plant was taken to Russia by Joffe when he left the works in 1948. In 1946/47, sodium hydroxide was produced at EKB electrolytically from sodium chloride, according to the Billider procedure. The sodium chloride contained up to three percent chlorine. Dechlorination was carried out in a vertically placed pipe (autoclave), the interior of which was equipped with a number of cylinder-shaped segments (called mushrooms in German nomenclature) arranged above each other. Ammonia was fed into the lower end of the pipe; sodium hydroxide was introduced into the upper end. The two chemicals would mix within the pipe. A valve at the bottom of the pipe released purified sodium hydroxide. The process had to be carried out with a temperature of 70° C, which was obtained by means of a heating spiral around the pipe, and under a pressure of no less than 40 atm. The process was not continuous. Sodium hydroxide and ammonia were fed into the pipe at intervals and then left to react upon each other. After reacting, the ammonia was released from the upper end of the pipe, purified of its sodium hydroxide contents by distillation, and re-used for the same purpose in a later stage of the process. The chlorine contents of the sodium hydroxide thus dechlorinated were between 0.03 and 0.05 percent. The Russian order of December 1952 specified that the 1946/47 development was to be repeated exactly, with the one exception that recovery and re-use of ammonia was to be eliminated. It further specified that the work had to be completed by 31 March 1953. Dr. Ehrhardt was again put in charge of the work, although he no longer belongs to the chemical section of the works. The work was completed on the requested date, with the exception of the research report which is to be delivered to the Russians; it is being prepared now.

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- b) Development of substitute contact matter to be used in the production of nitric acid through combustion of a mixture of 100 parts ammonia and 12 parts air. The works has used and is using now platinum screen contacts (Netzkontakte). These, however, are worn out and cannot be replaced easily. Experiments were carried out during the first four months of 1953 using cobalt oxalate and cobalt formate instead of platinum as contact matter. These experiments failed because the substitute catalysts, when exposed to the flow velocity of ammonia (500 cubic centimeter per second in a pipe with a diameter of 5 centimeter) disintegrated into a powder-like substance which was carried away by the ammonia. As of late April 1953, no decisive results had been obtained. The department is now attempting to find a means of stabilizing the substitute catalysts on a porous carrier in order to protect them against disintegration.
- c) Activation of silicic acid through pressure and temperature for its possible use as a catalyst. The works produce small amounts of silicic acid from phosphorus slacks for use in plants producing cleansing agents. In order to activate the silicic acid, some of it was made into a paste with water; the paste was put into an iron container and subjected for two to four hours to a pressure of 200 atm and a temperature of about 370° C. After drying, the silicic acid was poured into a glass tube and brought to a temperature of 400° C with the aid of a heating spiral around the tube. At this temperature the silicic acid showed catalytic properties. When methanol was blown through the tube, it separated into carbon monoxide and hydrogen through catalytic action of the silicic acid. The methanol was blown through the tube with nitrogen as its carrier gas. Other experiments showed that the silicic acid prepared in the way mentioned above acted as a catalyst with ethyl alcohol and formic acid. When attempts were made to determine the usefulness of activated silicic acid as a replacement for platinum contacts in the production of nitric acid, it turned out, however, that it could not be used for this purpose because its catalytic quality decreased in a relatively short time to such a point that it was no longer active. In order to overcome this shortcoming, the department planned to activate the silicic acid to a higher degree in an agitator container (Rührautoklav). As of early April 1953, such containers could not be procured in East Germany. The department is currently trying to rebuild its autoclave into an agitator autoclave with electromagnetic stirring. This work was begun at the end of April 1953.
- d) Experimental study of the Whirlpool procedure for catalysts. Although this is a 1953 priority order, work on it had not yet begun as of late April 1953. The department procured a prospectus of the US Dorr Company for use in the carrying out of this order.

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- 1/ ☐ Comment. Vofatit is also called levatit by the Bayer works, Leverkusen.
- 2/ ☐ Comment. The sales price is 980 DM per ton.
- 3/ ☐ Comment. Source could not specify the origin of the order more exactly. Presumably the selenic acid and the telluric acid were for a scientific institute in Russia.

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- 4/ ☐ Comment. Specifications for the purchased tellurium are not available.
- 5/ ☐ Comment. Source did not know exactly where in the Leuna plant the order originated.

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- 6/ ☐ Comment. Dr. Ehrhardt later resigned his position in the Main Laboratory and became the head of the SED organization of the plant. He also was made chairman of the German-Soviet Friendship Group.

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- 7/ ☐ Comment. The works were in possession of a photocopied US patent which served as basis for the work carried out in the department.

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